
पेट्रोलियम से व्युत्पन्न माइक्रोक्रीस्टलाइन
वैक्स — विशिष्टता
(पहला पुनरीक्षण)

Microcrystalline Wax Derived From
Petroleum — Specification
(First Revision)

ICS 75.140

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FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Petroleum and Their Related Products of Synthesis or Biological Origin Sectional Committee was approved by the Petroleum, Coal and Related Products Division Council.

This standard was originally published in 1993.

Petroleum origin microcrystalline waxes are imported for various end uses in the country. Recently, indigenous manufacturers are producing different types of microcrystalline wax from tank bottom wax. This standard was intended to assist in selecting and manufacturing suitable quality of the material for the right purpose.

In formulating this standard, assistance was derived from the following:

Petroleum waxes — Characterization performance and additives — The proceedings of the ASTM — TAPPI Symposium on petroleum waxes held at the 48th annual meeting of the Technical Association of the Pulp and Paper Industry February 18-21, 1963. Published data on Microcrystalline Wax of Petrolite Corporation, Bareco Division.

Characteristics of microcrystalline wax imported through marketing division of Indian Oil Corporation Limited (Data supplied by IOC (R and D) Centre, Faridabad).

In this revision, references and referenced test methods have been updated.

The composition of the Committee responsible for the formulation of this standard is given in Annex B.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

MICROCRYSTALLINE WAX DERIVED FROM PETROLEUM — SPECIFICATION

(*First Revision*)

1 SCOPE

This standard prescribes the requirements and method of sampling and tests for microcrystalline wax used for pharmaceuticals, cosmetics, packaging and various other industrial and general-purpose uses.

2 REFERENCES

The following standards contain provisions, which, through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards:

<i>IS No.</i>	<i>Title</i>
1070 : 1992	Water for general laboratory use (<i>third revision</i>)
1447 (Part 3) : 1992	Methods of sampling of petroleum and its products: Part 3 Methods of sampling of semi-solid and solid petroleum products (<i>first revision</i>)
1448	Methods of test for petroleum and its products
[P : 2] : 2007/ ISO 6619 : 1988	Petroleum products and lubricants — Neutralization number — Potentiometric titration method (<i>second revision</i>)
[P : 4/Sec 1] : 2008 / ISO 6245 : 2001	Petroleum products — Determination of ash (<i>second revision</i>)
[P : 12] : 2013/ ISO 2049 : 1996	Determination of colour ASTM scale (<i>second revision</i>)
[P : 25/Sec 1] : 2018/ ISO 3104 : 1994	Transparent and opaque liquids, Section 1 Determination of kinematic viscosity and calculation of dynamic viscosity (<i>second revision</i>)
[P : 52] : 2017/ ISO 2176 : 1995	Drop point (<i>second revision</i>)
[P : 55/Sec 1] : 2004	Determination of saponification value of petroleum products (<i>first revision</i>)

<i>IS No.</i>	<i>Title</i>
[P : 69] : 2013/ ISO 2592 : 2000	Determination flash and fire points — Cleveland open cup method (<i>first revision</i>)
[P : 93] : 1979	Needle penetration of petroleum waxes
2480 (Part 1) : 1983	General purpose glass thermometer: Part 1 Solid stem thermometers (<i>second revision</i>)
4654 : 1993	Paraffin wax — Specification (<i>second revision</i>)

3 TYPES AND GRADES

3.1 Types

The material shall be of the following five types:

- a) Type A — Low melting, for laminating and compounding with paraffin wax;
- b) Type B — Medium melting, for laminating and compounding with paraffin wax;
- c) Type C — Medium melting, for pharmaceutical and cosmetic trade;
- d) Type D — High melting, hard microcrystalline wax, for improving gloss and scuff, resistance of the new hot melt, wax- based copolymer blends; and
- e) Type E — High melting, hard, for various industrial and general purposes.

3.2 Grade

Subject to agreement between the purchaser and the supplier, each of the above five types shall be graded by its drop melting point of 2.5°C spread in the range of respective type.

4 REQUIREMENTS

4.1 Description

The material shall essentially be solid saturated hydrocarbons from petroleum, homogeneous in appearance and free from any foreign matter, objectionable odour and taste. It may contain only minimum specified liquid hydrocarbons and shall give on melting a clear liquid, free from water, dirt and other visible impurities.

4.2 The material shall also comply with the requirements given in Table 1 when tested according to the appropriate method prescribed under Annexes to this standard and 'P' series of IS 1448 reference to which is given in column 8 and 9 of the Table 1.

4.3 It is recognized that there are some applications where for technical or other reasons, limits different from those in this standard or additional requirements may be necessary. The standard does not cater to such special cases, which are for arrangement between the purchaser and the supplier.

5 PACKING AND MARKING

5.1 Packing

The microcrystalline waxes of types A, B, C and D shall be packed in polyethylene lined new gunny bags. For wax of type E new gunny bags or HDPE, woven sacks as agreed to between the purchaser and the supplier shall be used.

5.2 Marking

5.2.1 Each container shall be marked with the following information:

- Name, type, grade and mass of the material;
- Indication of the source of manufacture, recognized trademark, if any;
- Batch or code number or otherwise to enable the lot of consignment/or manufacture to be traced back from records;
- Melting point; and
- Container for type E shall be labelled conspicuously with the words:
"NOT TO BE USED FOR FOOD PACKAGING
COSMETICS PREPARATION OR MEDICINAL
USE"
- Any other statutory requirements.

Table 1 Requirement for Microcrystalline Wax of Petroleum Origin
(Clause 4.2)

Sl No.	Characteristics	Requirements for					Method of Test Reference to	
		Type A	Type B	Type C	Type D	Type E	Annex	[P :] of IS 1448
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)
i)	Drop melting point, °C	65-70	70-80	70-80	85-95	85-95	A	[P : 52]
ii)	Ash percent by mass, <i>Max</i> .	0.03	0.03	0.03	0.03	0.03	—	[P : 4/Sec 1]
iii)	Acidity:							
	a) Organic (mg of KOH per g), <i>Max</i>	0.10	0.10	0.10	0.10	0.10	—	[P : 2]
	b) Inorganic (mg of KOH per g), <i>Max</i>	0.02	0.02	0.02	0.02	0.03	—	—
iv)	Saponification value, <i>Max</i>	1.0	1.0	1.0	1.0	1.0	—	[P : 55/Sec 1]
v)	Oil content, percent by mass, <i>Max</i>	4.0	4.0	2.0	1.5	4.0	B of IS 4654	
vi)	Colour, ASTM, <i>Max</i>	2	2	0.5	1	5	—	[P : 12]
vii)	Needle penetration, 1/10 mm (at 25°, 5 s, 100 g)	10 to 25	20 to 35	25 to 35	2 to 10	2 to 10		[P : 93]
viii)	Viscosity at 100°C, mm ² /s	10 to 18	15 to 20	15 to 20	15 to 20	15 to 20		[P : 25/Sec 1]
ix)	Ultraviolet (UV absorbance per centimetre path length), <i>Max</i>						D of IS 4654	
	280-289 nm	0.15	0.15	0.15	0.15	—		
	290-299 nm	0.12	0.12	0.12	0.12	—		
	300-359 nm	0.08	0.08	0.08	0.08	—		
	360-400 nm	0.02	0.02	0.02	0.02	—		
x)	Flash point, COC, °C, <i>Min</i>	260	260	260	260	260	—	[P : 69]
xi)	Odour	0	0	0	0	0	E of IS 4654	

5.2.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

6 SAMPLING

6.1 The representative samples of the material shall be drawn as prescribed in IS 1447 (Part 3).

6.2 Tests on drop melting point shall be conducted on individual samples and the rest of the tests shall be conducted on composite samples.

ANNEX A

[Table 1, SI No. (i)]

DETERMINATION OF DROP MELTING POINT

A-1 GENERAL

This test method covers the determination of the drop melting point of petroleum wax. It is used primarily for petrolatums and other microcrystalline wax.

A-2 SIGNIFICANCE AND USE

Melting point is a property of wax that is, of interest to most wax consumers, it can be an indication of the performance properties of the wax. Drop melting point, test method IS 1448 [P : 52], is often used to measure the melting characteristics of petrolatums and other high viscosity petroleum waxes.

A-3 DEFINITION

A-3.1 Drop Melting Point of Petroleum Wax

The temperature at which material becomes sufficiently fluid to drop from the thermometer used in making the determination under definite prescribed conditions.

A-4 SUMMARY OF TEST METHOD

Specimens are deposited on two thermometer bulbs by dipping chilled thermometers into the sample. The thermometers bearing the specimens are placed in test tube and heated by means of a water bath until the specimens melt and the first drop falls from each thermometer bulb. The average of the temperatures at which these drops fall is the drop melting point of the sample.

A-5 APPARATUS

A-5.1 Test Tubes

Standard test tubes, 25 mm in outside diameter and 150 mm long. The test tube shall be supplied with corks grooved at the sides to permit air circulation and bored in the exact centre to receive the thermometer.

A-5.2 Bath

A transparent container of not less than 1 500 ml capacity, that will permit the immersion of the test tube to a depth of at least 90 mm and still leave a depth of 15 mm of water below the bottoms of the test tubes.

A-5.3 Thermometer

Having a range 32 to 127°C and least count of 0.2°C (*see Note*) inserted in a test tube with immersion length of 79 mm and having total length 381 mm.

NOTE — Thermometer of type ASTM 61C or IP 63C may be used.

A-5.4 Bath Thermometer

Any suitable type, accurate to 0.5°C throughout the required range.

A-6 PROCEDURE

Secure a sample of sufficient size that is representative of the material under inspection. Use a fresh portion of the sample for each set of two determinations. Melt the sample slowly until the temperature reached 93°C or about 11°C above the expected drop melting point, whichever is higher. Place sufficient sample in a flat bottom container to give a sample depth of 12 ± 1 mm. Adjust the temperature of the sample to 6 to 11°C (*see Note*) above its drop melting point using any general laboratory thermometer for measurement. Chill one of the test thermometer bulbs to 4°C. Wipe dry, and, quickly but carefully, immerse the chilled bulb vertically into the heated sample until it touches the bottom of the container (12 mm submerged) and withdraw it immediately. Hold the thermometer vertically away from the heat until the surface dulls, and then place it for 5 min in water having a temperature of 16°C. Prepare another sample specimen from the sample using this procedure.

NOTE — A dipping temperature of 11°C above the congealing point usually will be 6 to 11°C above the actual drop melting point.

Securely fix the thermometers in the test tubes by means of corks so that the tip of each thermometer is 15 mm above the bottom of its test tube. Insert the test tubes in the water bath, which is at 16°C, and adjust the height of the test tubes so that the immersion marks on the thermometers are level with the top surface of the water. Raise the temperature of the bath at the rate of 1.7°C/min to 38°C, then at a rate of 1°C/min until the first drop of material leaves each thermometer. Record in each case the temperature at which the first drop falls from the thermometer.

A-7 REPORT

Report the average of the two determinations as the drop melting point of the sample under test.

A-8 PRECISION AND BIAS

A-8.1 Precision

The precision of this test method as determined by statistical examination of inter-laboratory results is as follows:

A-8.1.1 Repeatability

The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed 0.8°C only in one case in twenty.

A-8.1.2 Reproducibility

The difference between two single and independent

results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed 1.3°C only in one case in twenty.

A-8.2 Bias

The procedure in this test method has no bias because the value of drop melting point can be defined only in terms of a test method.

ANNEX B*(Foreword)***COMMITTEE COMPOSITION**

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This Indian Standard has been developed from Doc No.: PCD 03 (13903).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Published by BIS, New Delhi